

**(12) UK Patent Application (19) GB (11) 2 195 649 (13) A**

(43) Application published 13 Apr 1988

(21) Application No **8720427**

(22) Date of filing **28 Aug 1987**

(30) Priority data

(31) **901484**

(32) **28 Aug 1986**

(33) **US**

(51) INT CL<sup>4</sup>

**C11D 1/14 1/29**

(52) Domestic classification (Edition J):

**C5D 6A5D2 6A5E 6A8B 6B11A 6B12B1 6B12F2 6B12G1  
6B12H 6B12M 6B13 6B2 6B4 6B5 6B7 6B8 6C8**

(56) Documents cited

**None**

(58) Field of search

**C5D**

**Selected US specifications from IPC sub-class C11D**

(71) Applicant

**Colgate-Palmolive Company**

**(Incorporated in USA-Delaware)**

**300 Park Avenue, New York, New York 10022, United  
States of America**

(72) Inventors

**Trazollah Ouhadi**

**Louis Dehan**

(74) Agent and/or Address for Service

**Kilburn & Stroda,**

**30 John Street, London WC1N 2DD**

**(54) Liquid nonionic laundry detergent composition containing higher alkyl sulphonate or alkyl ether  
sulphate stabilizer**

(57) A liquid heavy duty laundry detergent composition comprising a suspension of particles in liquid nonionic surfactant in which the stability against settling of the composition is increased by the addition of small amounts of a higher alkyl sulphonate or alkyl polyether sulphate. The yield stress of the compositions is improved with the same or lower plastic viscosity, especially at low concentrations of the alkyl sulphonate or alkyl polyether sulphate. The alkyl sulphonates and alkyl polyether sulphates also act to provide additional detergency.

**GB 2 195 649 A**

## SPECIFICATION

**Liquid nonionic laundry detergent composition containing higher alkyl sulphonate or alkyl ether sulphate stabilizer and method of use**

- 5 This invention relates to nonaqueous liquid fabric treating compositions. More particularly, this invention relates to nonaqueous liquid laundry detergent compositions which are stable against phase separation and gelation and are easily pourable and to the use of these compositions for cleaning soiled fabrics.
- 10 Liquid nonaqueous heavy duty laundry detergent compositions are well known in the art. For instance, compositions of that type may comprise a liquid nonionic surfactant in which are dispersed particles of a builder, as shown for instance in the U.S.P. Nos. 4,316,812, 3,630,929 and 4,264,466 and British patent Nos. 1,205,711, 1,270,040 and 1,600,981.
- 15 Liquid detergents are often considered to be more convenient to employ than dry powdered or particulate products and, therefore, have found substantial favour with consumers. They are readily measurable, speedily dissolved in the wash water, capable of being easily applied in concentrated solutions or dispersions to soiled areas on garments to be laundered and are nondusting, and they usually occupy less storage space. Additionally, the liquid detergents may have incorporated in their formulations materials which could not stand drying operations without
- 20 deterioration, which materials are often desirably employed in the manufacture of particulate detergent products. Although they are possessed of many advantages over unitary or particulate solid particles, liquid detergents often have certain inherent disadvantages too, which have to be overcome to produce acceptable commercial detergent products. Thus, some such products separate out on cooling and are not readily redispersed. In some cases the product viscosity
- 25 changes and it becomes either too thick to pour or so thin as to appear water. Some clear products become cloudy and others gel on standing.
- The present inventors have been involved in studying the behaviour of nonionic liquid surfactant systems with particulate matter suspended therein. Of particular interest has been nonaqueous built laundry liquid detergent compositions and the problem of settling of the suspended
- 30 builder and other laundry additives as well as the problem of gelling associated with nonionic surfactants. These considerations have an impact on, for example, product stability, pourability and dispersibility.
- It is known that one of the major problems with built liquid laundry detergents is their physical stability. This problem stems from the fact that the density of the solid particles dispersed in the
- 35 nonionic liquid surfactant is higher than the density of the liquid surfactant.
- Therefore, the dispersed particles tend to settle out. Two basic solutions exist to solve the settling out problem: increase nonionic liquid viscosity and reduce the dispersed solid particle size.
- It is known that suspensions can be stabilized against settling by adding inorganic or organic
- 40 thickening agents or dispersants, such as, for example, very high surface area inorganic materials, e.g. finely divided silica, clays, etc., organic thickeners, such as the cellulose ethers, acrylic and acrylamide polymers, polyelectrolytes, etc. However, such increases in suspensions viscosity are naturally limited by the requirement that the liquid suspension be readily pourable and flowable, even at low temperature. Furthermore, these additives do not contribute to the
- 45 cleaning performance of the formulation.
- Grinding to reduce the particle size provides the following advantages:
1. Specific surface area of the dispersed particles is increased, and, therefore, particle wetting by the nonaqueous vehicle (liquid nonionic) is proportionately improved.
  2. The average distance between dispersed particles is reduced with a proportionate increase
- 50 in particle-to-particle interaction. Each of these effects contributes to increase the rest-gel strength and the suspension yield stress while at the same time, grinding significantly reduces plastic viscosity.
- The yield stress is defined as the minimum stress necessary to induce a plastic deformation (flow) of the suspension. Thus, visualizing the suspension as a loose network of dispersed
- 55 particles, if the applied stress is lower than the yield stress, the suspension behaves like an elastic gel and no plastic flow will occur. Once the yield stress is overcome, the network breaks at some points and the sample begins to flow, but with a very high apparent viscosity. If the shear stress is much higher than the yield stress, the pigments are partially shear-deflocculated and the apparent viscosity decreases. Finally, if the shear stress is much higher than the yield
- 60 stress value, the dispersed particles are completely shear-deflocculated and the apparent viscosity is very low, as if no particle interaction were present.
- Therefore, the higher the yield stress of the suspension, the higher the apparent viscosity at low shear rate and the better is the physical stability against settling of the product.
- In addition to the problem of settling or phase separation the nonaqueous liquid laundry
- 65 detergents based on liquid nonionic surfactants suffer from the drawback that the nonionic tend

to gel when added to cold water. This is a particularly important problem in the ordinary use of European household automatic washing machines where the user places the laundry detergent composition in a dispensing unit (e.g. a dispensing drawer) of the machine. During the operation of the machine the detergent in the dispenser is subjected to a stream of cold water to transfer it to the main body of wash solution. Especially during the winter months when the detergent composition and water fed to the dispenser are particularly cold, the detergent viscosity increases markedly and a gel forms. As a result some of the composition is not flushed completely off the dispenser during operation of the machine, and a deposit of the composition builds up with repeated wash cycles, eventually requiring the user to flush the dispenser with hot water.

The gelling phenomenon can also be a problem whenever it is desired to carry out washing using cold water as may be recommended for certain synthetic and delicate fabrics or fabrics which can shrink in warm or hot water.

The tendency of concentrated detergent compositions to gel during storage is aggravated by storing the compositions in unheated storage areas, or by shipping the compositions during winter months in unheated transportation vehicles.

Partial solutions to the gelling problem have been proposed, for example, by diluting the liquid nonionic with certain viscosity controlling solvents and gel-inhibiting agents, such as lower alkanols, e.g. ethyl alcohol (see U.S.P. 3,953,380), alkali metal formates and adipates (see U.S.P. 4,368,147), hexylene glycol, polyethylene glycol, etc. and nonionic structure modification and optimization. As an example of nonionic surfactant modification one particularly successful result has been achieved by acidifying the hydroxyl moiety end group of the nonionic molecule. The advantages of introducing a carboxylic acid at the end of the nonionic include gel inhibition upon dilution; decreasing the nonionic pour point; and formation of an anionic surfactant when neutralized in the washing liquor. Nonionic structure optimization has centred on the chain length of the hydrophobic-lipophilic moiety and the number and make-up of alkylene oxide (e.g. ethylene oxide) units of the hydrophilic moiety. For example, it has been found that a C<sub>13</sub> fatty alcohol ethoxylated with 8 moles of ethylene oxide presents only a limited tendency to gel formation.

Nevertheless, improvements are desired in both the stability and gel inhibition of nonaqueous liquid fabric treating compositions.

In accordance with the present invention a highly concentrated stable nonaqueous liquid laundry detergent composition is prepared by adding to the composition small effective amounts of a higher alkyl sulphonate or higher alkyl polyether sulphate anti-settling stabilizing additive.

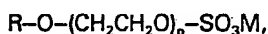
The compositions of the present invention contain as an essential ingredient a higher alkyl sulphonate or higher alkyl polyether sulphate anti-settling stabilizing additive. The anti-settling stabilizing additives are anionic surfactants. The anionic surfactants that are useful in the present invention are those surface active compounds which contain a long chain hydrocarbon hydrophobic group in their molecular structure and a hydrophile group, i.e. water solubilizing group such as sulphonate or sulphate group. The anionic surfactants include the water soluble higher alkyl sulphonates and the water soluble higher alkyl polyether sulphates.

The preferred anionic surface active agents are alkane, i.e. alkyl primary and secondary sulphonates, in which the alkyl group contains 8 to 26 carbon atoms, preferably 12 to 22 carbon atoms and more preferably 14 to 18 carbon atoms.

The alkyl sulphonates can be used as the alkali metal salts, such as sodium and potassium, the alkaline earth metal salts, such as calcium, magnesium and barium, and the ammonium cation salt. The preferred salts are the sodium and potassium salts.

The alkyl ether sulphates used as an anti-settling stabilizing additive in accordance with the present invention can be normal or branched chain alkyl and contain lower alkoxy groups, which can contain two or three carbon atoms.

The preferred alkyl poly lower alkoxy sulphates used in accordance with the present invention are the alkyl poly ethoxy sulphates represented by the formula



where R is a C<sub>8</sub>-C<sub>12</sub> alkyl, preferably C<sub>10</sub> to C<sub>18</sub> and more preferably C<sub>12</sub> to C<sub>15</sub>; p is 2 to 8, preferably 2 to 6, and more preferably 2 to 4; and M is an alkali metal, such as sodium and potassium, an alkaline earth metal, such as calcium, magnesium and barium and ammonium cation. The sodium and potassium salts are preferred.

In order to improve the viscosity characteristics of the composition an acid terminated nonionic surfactant can be added. To further improve the viscosity characteristics of the composition and the storage properties of the composition there can be added to the composition viscosity improving and anti-gel agents such as alkylene glycol mono alkyl ethers and additional anti-settling agents such as phosphoric acid ester and aluminium stearate. In preferred embodiments of the invention the detergent composition contains an acid terminated nonionic surfactant, an alkylene glycol mono alkyl ether, and an alkyl sulphonate or alkyl ether sulphate stabilizing anti-

settling agent.

Sanitizing or bleaching agents and activators therefore can be added to improve the bleaching and cleansing characteristics of the composition.

- 5 In an embodiment of the invention the builder components of the composition are ground to a particle size of less than 100 microns and to preferably less than 10 microns to further improve the stability of the suspension of the builder components in the liquid nonionic surfactant detergent. 5

In addition other ingredients can be added to the composition such as anti-incrustation agents, anti-foam agents, optical brighteners, enzymes, anti-redeposition agents, perfume and dyes.

- 10 The presently manufactured washing machines for home use normally operate at washing temperatures of up to 100°C. About up to 18 gallons (70 litres) of water are used during the wash and rinse cycles. 10

About 250 gms of power detergent per wash is normally used.

- 15 In accordance with the present invention where the highly concentrated liquid detergent is used normally only 100 gms (77 cc) of the liquid detergent composition is required to wash a full load of dirty laundry. 15

- Accordingly, in one aspect of the present invention there is provided a liquid heavy duty detergent composition composed of a suspension of a detergent builder salt in a liquid nonionic surfactant wherein the composition includes an amount of a higher alkyl sulphonate or a higher 20 alkyl polyether sulphate to increase the stability of the suspension against settling. 20

According to another aspect, the invention provides a concentrated liquid heavy duty laundry detergent composition which is stable, non-settling in storage and non-gelling in storage and in use. The liquid compositions of the present invention are easily pourable, easily measured and easily put into the washing machine.

- 25 According to another aspect, the invention provides a method for dispensing a liquid nonionic laundry detergent composition into and/or with cold water without undergoing gelation. In particular, a method is provided for filling a container with a nonaqueous liquid laundry detergent composition in which the detergent is composed, at least predominantly, of a liquid nonionic surface active agent and for dispensing the composition from the container into an 30 aqueous wash bath, wherein the dispensing is effected by directing a stream of unheated water onto the composition such that the composition is carried by the stream of water into the wash basin. 25

The addition of the higher alkyl sulphonates and higher alkyl polyether sulphates to the detergent compositions reduce the problem of dispersed particle settling and phase separation.

- 35 The concentrated nonaqueous liquid nonionic surfactant laundry detergent compositions of the present invention have the advantages of being stable, non-settling in storage, and non-gelling in storage. The liquid compositions are easily pourable, easily measured and easily put into the laundry washing machines. 35

- 40 This present invention provides heavy duty built nonaqueous liquid nonionic laundry detergent compositions which are non-gelling, stable suspensions and which include an effective amount of a higher alkyl sulphonate or a higher alkyl polyether sulphate which is sufficient to increase the yield stress of the composition to thereby increase its stability, i.e. prevent settling of builder particles, etc., preferably while reducing or at least without increasing, the plastic viscosity (viscosity under shear conditions) of the composition. 40

- 45 The detergent composition comprising a nonaqueous liquid nonionic surfactant and an effective amount of a higher alkyl polyether sulphate anti-settling agent sufficient to inhibit settling of the suspended particles, may include inorganic or organic fabric treating additives, e.g. viscosity improving and one or more anti-gel agents, anti-incrustation agents, pH control agents, bleaching agents, bleach activators, anti-foam agents, optical brighteners, enzymes, anti-redeposition 50 agents, perfume and dyes. 50

- 55 In accordance with the present invention the physical stability of the suspension of the detergent builder compound or compounds and any other suspended additive, such as bleaching agent, etc. in the liquid nonionic surfactant vehicle is substantially improved by the addition of an anti-settling stabilizing agent which is an anionic surfactant higher alkyl sulphonate or an anionic surfactant higher alkyl poly ether sulphate. 55

The addition of very small amounts of the anionic surfactant anti-settling stabilizing agents is sufficient to substantially improve the physical stability of the detergent compositions.

The anionic higher alkyl sulphonate and higher alkyl polyether sulphate surfactants are commercially available and/or can readily be manufactured by known procedures.

- 60 The compositions of the present invention contain as an essential ingredient a higher alkyl sulphonates or a higher alkyl polyether anti-settling stabilizing additive. The anti-settling stabilizing additive can comprise one or more anionic surface active agents. 60

- 65 The anionic surface active agents that are useful in the present invention are those surface active compounds which contain a long chain hydrocarbon hydrophobic group in their molecular structure and a hydrophile group, i.e. water solubilizing group such as sulphonate or sulphate 65

group.

The anionic surface active agents include the water soluble higher alkyl sulphonates and the water soluble higher alkyl poly ether sulphates.

The preferred anionic surface active agents are alkane, i.e. higher alkyl primary and secondary sulphonates, in which the alkyl group contains 8 to 26 carbon atoms, preferably 12 to 22 carbon atoms and more preferably 14 to 18 carbon atoms. 5

The primary and secondary alkyl sulphonates can be made by reacting long chain alpha-olefins with sulphites or bisulphites, e.g. sodium bisulphite.

The alkyl sulphonates can also be made by reacting longchain normal paraffin hydrocarbons with sulphur dioxide and oxygen as described in U.S.P. Nos. 2,503,280, 2,507,088, 3,372,188 and 3,260,741 to obtain normal or secondary higher alkyl sulphonates suitable for use as surfactant detergents. 10

The alkyl substituent is preferably linear, i.e. normal alkyl, however, branched chain alkyl sulphonates can be employed, although they are not as good with respect to biodegradability. The alkane, i.e. alkyl, substituent may be terminally sulphonated or may be joined to the 2-carbon atom of the chain, i.e. may be a secondary sulphonate. 15

The higher alkyl sulphonates can be used as the alkali metal salts, such as sodium and potassium, an alkaline earth metal salt, such as calcium, magnesium and barium, and the ammonium cation salt. The preferred salts are the sodium and potassium salts.

The more preferred alkyl sulphonates are the C<sub>14</sub> to C<sub>18</sub> primary normal alkyl sodium and potassium sulphonates, with the C<sub>15</sub> primary normal alkyl sodium sulphonate salt being the most preferred. The C<sub>15</sub> normal primary alkyl sodium sulphonate salt is sold under the tradename Mersolat H 98 (C15). 20

The higher alkyl sulphonates are generally more effective than the higher alkyl polyether sulphonates. 25

Mixtures of higher alkyl sulphonates can be used as well as mixtures of higher alkyl sulphonates and higher alkyl poly ether sulphates.

The higher alkyl polyether sulphates used as an anti-settling stabilizing additive in accordance with the present invention can be normal or branched chain alkyl and contain lower alkoxy group which can contain two or three carbon atoms. The normal higher alkyl polyether sulphates are preferred in that they have a higher degree of biodegradability than the branched chain alkyl and the lower poly alkoxy groups are preferably ethoxy groups. 30

The preferred higher alkyl poly ethoxy sulphates used in accordance with the present invention are represented by the formula

$$R^1-O(CH_2CH_2O)_p-SO_3M,$$
 35

wherein R<sup>1</sup> is a C<sub>8</sub> to C<sub>20</sub> alkyl, preferably 2 to 6, and more preferably 2 to 4; and M is an alkali metal, such as sodium and potassium, an alkaline earth metal, such as calcium, magnesium and barium, or ammonium cation. The sodium and potassium salts are preferred. 40

A preferred higher alkyl poly ethoxylated sulphate is the sodium salt of a triethoxy C<sub>12</sub> to C<sub>15</sub> alcohol sulphate having the formula

$$C_{12-15}-O-(CH_2CH_2O)_3-SO_3Na.$$
 45

Examples of other suitable higher alkyl poly lower alkoxy sulphates that can be used in accordance with the present invention are C<sub>12-15</sub> normal or primary alkyl triethenoxy sulphate, sodium salt; n-decyl diethenoxy sulphate, sodium salt; C<sub>12</sub> primary alkyl diethenoxy sulphate, ammonium salt; C<sub>15</sub> primary alkyl tetraethenoxy sulphate, sodium salt; mixed C<sub>14-15</sub> normal primary alkyl mixed tri- and tetraethenoxy sulphate, sodium salt; stearyl pentaethenoxy sulphate, sodium salt; and mixed C<sub>10-18</sub> normal primary alkyl triethenoxy sulphate, potassium salt. 50

The normal alkyl poly-lower alkoxy sulphates are readily biodegradable and are preferred. The alkyl poly-lower alkoxy sulphates can be used in mixtures with each other and/or in mixtures with the above discussed higher alkyl sulphonates.

Although applicants do not wish to be bound by any particularly theory of the manner by which the anionic surfactants function to prevent settling of the suspended particles, it is presumed that the alkyl sulphonates or alkyl ether sulphates increase the wettability of the dispersed solid particle surfaces by the nonionic surfactant. This increase in wettability, therefore, allows the suspended particles to more easily remain in suspension. 55

The increased physical stability is manifested by an increase in the yield stress of the composition by as much as about 100% or more, for example, in the case of Mersolat H98(C15) from 2 Pa to 4 Pa, as compared to the same composition without the stabilizing agent. As described above, the higher is the yield stress, the higher is the apparent viscosity at low shear rate and the better is the physical stability. 60

Only very small amounts of the higher alkyl sulphonates or higher alkyl polyether sulphates 65

stabilizing agent is required to obtain the significant improvements in physical stability. For example, based on the total weight of the nonionic liquid surfactant composition, suitable amounts of the higher alkyl sulphonate or higher alkyl polyether sulphate are in the range of from about 0.1% to about 5%, preferably from about 0.3% to about 2.0% and more preferably about 0.5 to 1.5%.

In addition to its action as a physical stabilizing agent, the higher alkyl sulphonates and higher alkyl polyether sulphates have the additional advantages over other physical stabilizing agents that they are anionic in character and are compatible with the nonionic surfactant component and exhibit some detergent activity.

10 While the higher alkyl sulphonates or higher alkyl polyether sulphates alone are effective in their physical stabilizing action, there can be added to the formulation other known physical stabilizers, such as, for example, an acidic organic phosphorus compound having an acidic-POH group, such as a partial ester of phosphorous acid and an alkanol or an aluminium salt of a fatty acid.

15 The nonionic synthetic organic detergents employed in the practice of the invention may be any of a wide variety of known compounds.

As is well known, the nonionic synthetic organic detergents are characterised by the presence of an organic hydrophobic group and an organic hydrophilic group and are typically produced by the condensation of an organic aliphatic or alkyl aromatic hydrophobic compound with ethylene oxide (hydrophilic in nature). Practically any hydrophobic compound having a carboxy, hydroxy, amido or amino group with a free hydrogen attached to the nitrogen can be condensed with ethylene oxide or with the polyhydration product thereof, polyethylene glycol, to form a nonionic detergent. The length of the hydrophilic or polyoxy ethylene chain can be readily adjusted to achieve the desired balance between the hydrophobic and hydrophilic groups. Typical suitable

25 nonionic surfactants are those disclosed in U.S. Patents 4,316,812 and 3,630,929.

Usually, the nonionic detergents are poly-lower alkoxyated lipophiles wherein the desired hydrophilelipophile balance is obtained from addition of a hydrophilic poly-lower alkoxy group to a lipophilic moiety. A preferred class of the nonionic detergent employed is the poly-lower alkoxyated higher alkanol wherein the alkanol is of 9 to 18 carbon atoms and wherein the

30 number of mols of lower alkylene oxide (of 2 or 3 carbon atoms) is from 3 to 12. Of such materials it is preferred to employ those wherein the higher alkanol is a higher fatty alcohol of 9 to 11 or 12 to 15 carbon atoms and which contain from 5 to 8 or 5 to 9 lower alkoxy groups per mol. Preferably, the lower alkoxy is ethoxy but in some instances, it may be desirably mixed with propoxy, the latter, if present, often being a minor (less than 50%) proportion.

35 Exemplary of such compounds are those wherein the alkanol is of 12 to 15 carbon atoms and which contain about 7 ethylene oxide groups per mol, e.g. Neodol 25-7 and Neodol 23-6.5, which products are made by Shell Chemical Company, Inc. The former is a condensation product of a mixture of higher fatty alcohols averaging about 12 to 15 carbon atoms, with about 7 mols of ethylene oxide and the latter is a corresponding mixture wherein the carbon atom content of

40 the higher fatty alcohol is 12 to 13 and the number of ethylene oxide groups present averages about 6.5. The higher alcohols are primary alkanols.

Other examples of such detergents include Tergitol 15-S-7 and Tergitol 15-S-9, both of which are linear secondary alcohol ethoxylates made by Union Carbide Corp. The former is mixed ethoxylation product of 11 to 15 carbon atoms linear secondary alkanol with seven mols of ethylene oxide and the latter is a similar product but with nine mols of ethylene oxide being reacted.

Also useful in the present composition as a component of the nonionic detergent are higher molecular weight nonionics, such as Neodol 45-11, which are similar ethylene oxide condensation products of higher fatty alcohols, with the higher fatty alcohol being of 14 to 15 carbon atoms and the number of ethylene oxide groups per mol being about 11. Such products are also made by Shell Chemical Company.

50 Other useful nonionics are represented by the commercially well known class of nonionics sold under the trademark Plurafac. The Plurafacs are the reaction product of a higher linear alcohol and a mixture of ethylene and propylene oxides, containing a mixed chain of ethylene oxide and propylene oxide, terminated by a hydroxyl group. Examples include Product A (a C<sub>13</sub>-C<sub>15</sub> fatty alcohol condensed with 6 moles ethylene oxide and 3 moles propylene oxide), Product B (a C<sub>13</sub>-C<sub>15</sub> fatty alcohol condensed with 7 moles propylene oxide and 4 moles ethylene oxide), Product C (a C<sub>13</sub>-C<sub>15</sub> fatty alcohol condensed with 5 moles propylene oxide and 10 moles ethylene oxide, Plurafac B26), and Product D (a mixture of equal parts Product C and Product B).

60 Another group of liquid nonionics are commercially available from Shell Chemical Company, Inc. under the Dobanol trademark: Dobanol 91-5 is an ethoxylated C<sub>9</sub>-C<sub>11</sub> fatty alcohol with an average of 5 moles ethylene oxide and Dobanol 25-7 is an ethoxylated C<sub>12</sub>-C<sub>15</sub> fatty alcohol with an average of 7 moles ethylene oxide per mole of fatty alcohol.

In the preferred poly-lower alkoxyated higher alkanols, to obtain the best balance of hydrophilic and lipophilic moieties the number of lower alkoxyes will usually be from 40 to 100% of

the number of carbon atoms in the higher alcohol, preferably 40 to 60% thereof and the nonionic detergent will preferably contain at least 50% of such preferred poly-lower alkoxy higher alkanol. Higher molecular weight alkanols and various other normally solid nonionic detergents and surface active agents may be contributory to gelation of the liquid detergent and consequently, will preferably be omitted or limited in quantity in the present compositions, although minor proportions thereof may be employed for their cleaning properties, etc. With respect to both preferred and less preferred nonionic detergents the alkyl groups present therein are generally linear although branching may be tolerated, such as at a carbon next to or two carbons removed from the terminal carbon of the straight chain and away from the ethoxy chain, if such branched alkyl is not more than three carbons in length. Normally, the proportion of carbon atoms in such a branched configuration will be minor rarely exceeding 20% of the total carbon atom content of the alkyl. Similarly, although linear alkyls which are terminally joined to the ethylene oxide chains are highly preferred and are considered to result in the best combination of detergency, biodegradability and non-gelling characteristics, medial or secondary joinder to the ethylene oxide in the chain may occur. It is usually in only a minor proportion of such alkyls, generally less than 20% but, as is in the cases of the mentioned Tergitols, may be greater. Also, when propylene oxide is present in the lower alkylene oxide chain, it will usually be less than 20% thereof and preferably less than 10% thereof.

When greater proportions of non-terminally alkoxyated alkanols, propylene oxide-containing poly-lower alkoxyated alkanols and less hydrophile-lipophile balanced nonionic detergent than mentioned above are employed and when other nonionic detergents are used instead of the preferred nonionics recited herein, the product resulting may not have as good detergency, stability, viscosity and non-gelling properties as the preferred compositions but use of the viscosity and gel controlling compounds of the invention can also improve the properties of the detergents based on such nonionics. In some cases, as when a higher molecular weight poly-lower alkoxyated higher alkanol is employed, often for its detergency, the proportion thereof will be regulated or limited in accordance with the results of routine experiments, to obtain the desired detergency and still have the product non-gelling and of desired viscosity. Also, it has been found that it is only rarely necessary to utilize the higher molecular weight nonionics for their detergent properties since the preferred nonionics described herein are excellent detergents and additionally, permit the attainment of the desired viscosity in the liquid detergent without gelation at low temperatures.

Another useful group of nonionic surfactants are the "Surfactant T" series of nonionics available from British Petroleum. The Surfactant T nonionics are obtained by the ethoxylation of secondary  $C_{13}$  fatty alcohols having a narrow ethylene oxide distribution. The Surfactant T5 has an average of 5 moles of ethylene oxide; Surfactant T7 an average of 7 moles of ethylene oxide; Surfactant T9 an average of 9 moles of ethylene oxide and Surfactant T12 an average of 12 moles of ethylene oxide per mole of secondary  $C_{13}$  fatty alcohol.

In the compositions of this invention, preferred nonionic surfactants include the  $C_{12}$ - $C_{16}$  secondary fatty alcohols with relatively narrow contents of ethylene oxide in the range of from about 7 to 9 moles, and the  $C_9$  to  $C_{11}$  fatty alcohols ethoxylated with about 5-6 moles ethylene oxide.

Mixtures of two or more of the liquid nonionic surfactants can be used and in some cases advantages can be obtained by the use of such mixtures.

The viscosity and gel properties of the liquid detergent compositions can be improved by including in the composition an effective amount of an acid terminated liquid nonionic surfactant. The acid terminated nonionic surfactants consist of a nonionic surfactant which has been modified to convert a free hydroxyl group thereof to a moiety having a free carboxyl group, such as an ester or a partial ester of a nonionic surfactant and a polycarboxylic acid or anhydride.

As disclosed in the commonly assigned copending US Application Serial No. 597,948 filed April 9, 1984, corresponding to GB Application No. 8509084 Serial No. 2158454, the disclosure of which is incorporated herein by reference, the free carboxyl group modified nonionic surfactants, which may be broadly characterised as polyether carboxylic acids, function to lower the temperature at which the liquid nonionic forms a gel with water.

The addition of the acid terminated nonionic surfactants to the liquid nonionic surfactant aids in the dispensibility of the composition, i.e. pourability, and lowers the temperature at which the liquid nonionic surfactants form a gel in water without a decrease in their stability against settling. The acid terminated nonionic surfactant reacts in the washing machine water with the alkalinity of the dispersed builder salt phase of the detergent composition and acts as an effective anionic surfactant.

Specific examples include the half-esters of Plurafact RA30 with succinic anhydride, the ester or half ester of Dobanol 25-7 with succinic anhydride, and the ester or half ester of Dobanol 91-5 with succinic anhydride. Instead of succinic anhydride, other polycarboxylic acids or anhydrides can be used, e.g. maleic acid, maleic acid anhydride, glutaric acid, malonic acid, phthalic acid, phthalic anhydride, citric acid and the like.

The acid terminated nonionic surfactants can be prepared as follows:



Acid Terminated Product A. 400 g of Product A nonionic surfactant which is a C<sub>13</sub> to C<sub>15</sub> alkanol which has been alkoxylated to introduce 6 ethylene oxide and 3 propylene oxide units per alkanol unit is mixed with 32 g of succinic anhydride and heated for 7 hours at 100°C. The mixture is cooled and filtered to remove unreacted succinic material. Infrared analysis indicated that about one half of the nonionic surfactant has been converted to the acidic half-ester thereof.

Acid Terminated Dobanol 25-7. 522 g of Dobanol 25-7 nonionic surfactant which is the product of ethoxylation of a C<sub>12</sub> to C<sub>15</sub> alkanol and has about 7 ethylene oxide units per molecule of alkanol is mixed with 100 g of succinic anhydride and 0.1 g of pyridine (which acts as an esterification catalyst) and heated at 260°C for 2 hours, cooled and filtered to remove unreacted succinic material. Infrared analysis indicates that substantially all the free hydroxyls of the surfactant have reacted.

Acid Terminated Dobanol 91-5. 1000 g of Dobanol 91-5 nonionic surfactant which is the product of ethoxylation of a C<sub>9</sub> to C<sub>11</sub> alkanol and has about 5 ethylene oxide units per molecule of alkanol is mixed with 265 g of succinic anhydride and 0.1 g of pyridine catalyst and heated at 260°C for 2 hours, cooled and filtered to remove unreacted succinic material. Infrared analysis indicates that substantially all the free hydroxyls of the surfactant have reacted.

Other esterification catalysts, such as an alkali metal alkoxide (e.g. sodium methoxide) may be used in place of, or in admixture with, the pyridine.

The acidic polyether compound, i.e. the acid terminated nonionic surfactant is preferably added dissolved in the nonionic surfactant.

The liquid nonaqueous nonionic surfactant used in the compositions of the present invention has dispersed and suspended therein fine particles of organic and/or inorganic detergent builder salts.

The detergent compositions of the present invention include water soluble and/or water insoluble detergent builder salts. Water soluble inorganic alkaline builder salts which can be used alone with detergent compound or in admixture with other builders are alkali metal carbonates, bicarbonates, borates, phosphates, polyphosphates, and silicates. (Ammonium or substituted ammonium salts can also be used.) Specific examples of such salts are sodium tripolyphosphate, sodium carbonate, sodium tetraborate, sodium pyrophosphate, potassium pyrophosphate, sodium bicarbonate, potassium tripolyphosphate, sodium hexametaphosphate, sodium sesquicarbonate, sodium mono and diorthophosphate, and potassium bicarbonate. Sodium tripolyphosphate (TPP) is especially preferred.

Since the compositions of this invention are generally highly concentrated, and, therefore, may be used at relatively lower dosages, it is desirable to supplement any phosphate builder (such as sodium tripolyphosphate) with an auxiliary builder such as a poly lower carboxylic acid or a polymeric carboxylic acid having high calcium binding capacity to inhibit incrustation which could otherwise be caused by formation of an insoluble calcium phosphate.

A suitable lower poly carboxylic acid comprises alkali metal salts of lower polycarboxylic acids, preferably the sodium and potassium salts. Suitable lower polycarboxylic acids have two to four carboxylic acid groups. The preferred sodium and potassium lower polycarboxylic acid salts are the citric and tartaric acid salts.

The sodium citric acid salts are the most preferred, especially the trisodium citrate. The monosodium and disodium citrates can also be used. The monosodium and disodium tartaric acid salts can also be used. The alkali metal lower polycarboxylic acid salts are particularly good builder salts; because of their high calcium and magnesium binding capacity they inhibit incrustation which could otherwise be caused by formation of insoluble calcium and magnesium salts.

Other organic builders are polymers and copolymers of polyacrylic acid and polymaleic anhydride and the alkali metal salts thereof. More specifically such builder salts can consist of a copolymer which is the reaction product of about equal moles of methacrylic acid and maleic anhydride which has been completely neutralized to form the sodium salt thereof. The builder is commercially available under the tradename of Sokalan CP5. This builder serves when used even in small amounts to inhibit incrustation.

Examples of organic alkaline sequestrant builder salts which can be used with the detergent builder salts or in admixture with other organic and inorganic builders are alkali metal, ammonium or substituted ammonium, aminopolycarboxylates, e.g. sodium and potassium ethylene diamine-tetracetate (EDTA), sodium and potassium nitrilotriacetates (NTA), and triethanolammonium N—(2-hydroxy thyl)nitrilotriacetates. Mixed salts of these aminopolycarboxylates are also suitable.

Other suitable builders of the organic type include carboxymethylsuccinates, tartronates and glycollates. Of special value are the polyacetal carboxylates. The polyacetal carboxylates and their use in detergent compositions are described in US Application Serial No. 767,570, filed August 19, 1985, corresponding to GB Application No. 8619793 Serial No. 2179365 assigned to applicants' assignee and in U.S.P. Nos. 4,144,226, 4,315,092 and 4,146,495.

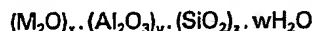
The alkali metal silicates are useful builder salts which also function to adjust or control the pH and to make the composition anticorrosive to washing machine parts. Sodium silicates of Na<sub>2</sub>O/SiO<sub>2</sub> ratios of from 1.6/1 to 1/3.2, especially about 1/2 to 1.2.8 are preferred. Potassium



silicates of the same ratios can also be used.

Other typical suitable builders include, for example, those disclosed in U.S. Patents 4,316,812, 4,264,466 and 3,630,929. The inorganic builder salts can be used with the nonionic surfactant detergent compound or in admixture with other inorganic builder salts or with organic builder salts.

The water insoluble crystalline and amorphous aluminosilicate zeolites can be used. The zeolites generally have the formula



wherein x is 1, y is from 0.8 to 1.2 and preferably 1, z is from 1.5 to 3.5 or higher and preferably 2 to 3 and w is from 0 to 9, preferably 2.5 to 6 and M is preferably sodium. A typical zeolite is type A or similar structure, with type 4A particularly preferred. The preferred aluminosilicates have calcium ion exchange capacities of about 200 milliequivalents per gram or greater, e.g. 400 meq/g.

Various crystalline zeolites (i.e. aluminosilicates) that can be used are described in British Patent 1,504,168, U.S.P. 4,409,136 and Canadian Patents 1,072,835 and 1,087,477, all of which are hereby incorporated by reference for such descriptions. An example of amorphous zeolites useful herein can be found in Belgium Patent 835,351 and this patent too is incorporated herein by reference.

Other materials such as clays, particularly of the water-insoluble types, may be useful adjuncts in compositions of this invention. Particularly useful is bentonite. This material is primarily montmorillonite which is a hydrated aluminium silicate in which about 1/6th of the aluminium atoms may be replaced by magnesium atoms and with which varying amounts of hydrogen, sodium, potassium, calcium, etc., may be loosely combined. The bentonite in its more purified form (i.e. free from any grit, sand, etc.) suitable for detergents contains at least 50% montmorillonite and thus its cation exchange capacity is at least about 50 to 75 meq per 100 g of bentonite. Particularly preferred bentonites are the Wyoming or Western U.S. bentonites which have been sold as Thixo-jels 1, 2, 3 and 4 by Georgia Kaolin Co. These bentonites are known to soften textiles as described in British Patent 401,413 to Marriott and British Patent 461,221 to Marriott and Duggan.

The inclusion in the detergent composition of an effective amount of low molecular weight amphiphilic compounds which function as viscosity control and gel-inhibiting agents for the nonionic surfactant substantially improves the storage properties of the composition. The amphiphilic compounds can be considered to be analogous in chemical structure to the ethoxylated and/or propoxylated fatty alcohol liquid nonionic surfactants but have relatively short hydrocarbon chain lengths ( $C_2$  to  $C_6$ ) and a low content of ethylene oxide (about 2 to 6 ethylene oxide groups per molecule).

Suitable amphiphilic compounds can be represented by the following general formula



where R is a  $C_2$ - $C_8$  alkyl group, and n is a number of from about 1 to 6, on average.

Specifically the compounds are lower ( $C_2$  to  $C_3$ ) alkylene glycol mono lower ( $C_2$  to  $C_6$ ) alkyl ethers.

More specifically the compounds are mono di- or tri-lower ( $C_2$  to  $C_3$ ) alkylene glycol mono lower ( $C_2$  to  $C_6$ ) alkyl ethers.

Specific examples of suitable amphiphilic compounds include

ethylene glycol monoethyl ether ( $C_2H_5-O-CH_2CH_2OH$ ),

diethylene glycol monobutyl ether ( $C_4H_9-O-(CH_2CH_2O)_2H$ ),

tetraethylene glycol monobutyl ether

( $C_4H_9-O-(CH_2CH_2O)_4H$ ) and

dipropylene glycol monomethyl ether

$CH_3-O-(CH_2CHO)_2H$ .



Diethylene glycol monobutyl ether is especially preferred.

The inclusion in the composition of the low molecular weight lower alkylene glycol mono alkyl ether decreases the viscosity of the composition, such that it is more easily pourable, improves the stability against settling and improves the dispersibility of the composition on the addition to warm water or cold water.

The compositions of the present invention have improved viscosity and stability characteristics and remain stable and purable at temperatures as low as about 5°C and lower.

In the embodiment of this invention a supplemental stabilizing agent which is an alkanol ester of phosphoric acid or an aluminium salt of a higher fatty acid can be added to the formulation.

Improvements in stability of the composition may be achieved by incorporation of a small effective amount of an acidic organic phosphorus compound having an acidic -POH group, such as a partial ester of phosphorous acid and an alkanol.

As disclosed in the commonly assigned copending US Application Serial No. 597,948 filed April 9, 1984, corresponding to GB Application No. 8509024 Serial No. 2158454, the disclosure of which is incorporated herein by reference, the acidic organic phosphorus compound having an acidic -POH group can increase the stability of the suspension of builders in the nonaqueous liquid nonionic surfactant.

The acidic organic phosphorus compound may be, for instance, a partial ester of phosphoric acid and an alcohol such as an alkanol which has a lipophilic character, having, for instance, more than 5 carbon atoms, e.g. 8 to 20 carbon atoms.

A specific example is a partial ester of phosphoric acid and a C<sub>18</sub> to C<sub>18</sub> alkanol (Empiphos 5632 from Marchon); it is made up of about 35% monoester and 65% diester.

The inclusion of quite small amounts of the acidic organic phosphorus compound makes the suspension stable against settling on standing but remains pourable, while, for the low concentration of stabilizer, e.g. below about 1%, its plastic viscosity will generally decrease.

Improvements in the stability and anti-settling properties of the composition may be achieved by the addition of a small effective amount of an aluminium salt of a higher fatty acid to the composition.

The aluminium salt stabilizing agents are the subject matter of the commonly assigned US copending application Serial No. 725,455, filed April 22, 1985, corresponding to GB Application No. 8604969, Serial No. 2172897, the disclosure of which is incorporated herein by reference.

The preferred higher aliphatic fatty acids will have from about 8 to about 22 carbon atoms, more preferably from about 10 to 20 carbon atoms, and especially preferably from about 12 to 18 carbon atoms. The aliphatic radical may be saturated or unsaturated and may be straight or branched. As in the case of the nonionic surfactants, mixtures of fatty acids may also be used, such as those derived from natural sources, such as tallow fatty acid, coco fatty acid, etc.

Examples of the fatty acids from which the aluminium salt stabilizers can be formed include, decanoic acid, dodecanoic acid, palmitic acid, myristic acid, stearic acid, oleic acid, eicosanoic acid, tallow fatty acid, coco fatty acid, mixtures of these acids, etc. The aluminium salts of these acids are generally commercially available, and are preferably used in the triacid form, e.g. aluminium stearate as aluminium tristearate  $\text{Al}(\text{C}_{17}\text{H}_{35}\text{COO})_3$ . The monoacid salts, e.g. aluminium monostearate,  $\text{Al}(\text{OH})(\text{C}_{17}\text{H}_{35}\text{COO})$  and diacid salts, e.g. aluminium distearate,  $\text{Al}(\text{OH})_2(\text{C}_{17}\text{H}_{35}\text{COO})_2$ , and mixtures of two or three of the mono-, di- and triacid aluminium salts can also be used. It is most preferred, however, that the triacid aluminium salt comprises at least 30%, preferably at least 50%, especially preferably at least 80% of the total amount of aluminium fatty acid salt.

The aluminium salts, as mentioned above, are commercially available and can be easily produced by, for example, saponifying a fatty acid, e.g. animal fat, stearic acid, etc., followed by treatment of the resulting soap with alum, alumina, etc.

Only very small amounts of the aluminium salt stabilizing agent is required to obtain an improvement in physical stability.

The bleaching agents are classified broadly, for convenience, as chlorine bleaches and oxygen bleaches. Chlorine bleaches are typified by sodium hypochlorite ( $\text{NaOCl}$ ), potassium dichloroisocyanurate (59% available chlorine), and trichloroisocyanuric acid (95% available chlorine). Oxygen bleaches are preferred and are represented by percompounds which liberate hydrogen peroxide in solution. Preferred examples include sodium and potassium perborates, percarbonates, and phosphates, and potassium monopersulphate. The perborates, particularly sodium perborate monohydrate, are especially preferred.

The peroxygen compound is preferably used in admixture with an activator therefor. Suitable activators which can lower the effective operating temperature of the peroxide bleaching agent are disclosed, for example, in U.S.P. 4,264,466 or in column 1 of U.S.P. 4,430,244, the relevant disclosures of which are incorporated herein by reference. Polyacylated compounds are preferred activators; among these, compounds such as tetraacetyl ethylene diamine ("TAED") and pentaacetyl glucose are particularly preferred. Other useful activators include, for example, acetylsalicylic acid derivatives, ethylidene benzoate acetate and its salts, ethylidene carboxylate acetate and its salts, alkyl and alkenyl succinic anhydride, tetraacetylglucouril ("TAGU"), and the derivatives of these. Other useful classes of activators are disclosed, for example, in U.S.P. 4,111,826, 4,422,950 and 3,661,789.

The bleach activator usually interacts with the peroxygen compound to form a peroxyacid bleaching agent in the wash water. It is preferred to include a sequestering agent of high complexing power to inhibit any undesired reaction between such peroxyacid and hydrogen peroxide in the wash solution in the presence of metal ions.

Suitable sequestering agents for this purpose include the sodium salts of nitrilotriacetic acid (NTA), ethylene diamine tetraacetic acid (EDTA), diethylene triamine pentaacetic acid (DTPA), diethylene triamine pentamethylene phosphonic acid (DTPMP) sold under the tradename Dequest

2066; and ethylene diamine tetramethylene phosphonic acid (EDITEMPA). The sequestering agents can be used alone or in admixture.

In order to avoid loss of peroxide bleaching agent, e.g. sodium perborate, resulting from enzyme-induced decomposition, such as by catalase enzyme, the compositions may additionally include an enzyme inhibitor compound, i.e. a compound capable of inhibiting enzyme-induced decomposition of the peroxide bleaching agent. Suitable inhibitor compounds are disclosed in U.S.P. 3,606,990, the relevant disclosure of which is incorporated herein by reference.

Of special interest as the inhibitor compound, mention can be made of hydroxylamine sulphate and other water-soluble hydroxylamine salts. In the preferred nonaqueous compositions of this invention, suitable amounts of the hydroxylamine salt inhibitors can be as low as about 0.01 to 0.4%. Generally, however, suitable amounts of enzyme inhibitors are up to about 15%, for example, 0.1 to 10%, by weight of the composition.

In addition to the detergent builders, various other detergent additives or adjuvants may be present in the detergent product to give it additional desired properties, either of functional or aesthetic nature. Thus, there may be included in the formulation, minor amounts of soil suspending or anti-redeposition agents, e.g. polyvinyl alcohol, fatty amides, sodium carboxymethyl cellulose, hydroxy-propyl methyl cellulose. A preferred anti-redeposition agent is sodium carboxymethyl cellulose having a 2:1 ratio of CM/MC which is sold under the tradename Relatin DM 4050.

Optical brighteners for cotton, polyamide and polyester fabrics can be used. Suitable optical brighteners include stilbene, triazole and benzidine sulphone compositions, especially sulphonated substituted triazinyl stilbene, sulphonated naphthotriazole stilbene, benzidine sulphone, etc.; most preferred are stilbene and triazole combinations. A preferred brightener is Stilbene Brightener N4 which is a dimorpholine dianilino stilbene sulphonate.

Enzymes, preferably proteolytic enzymes, such as subtilisin, bromelin, papain, trypsin and pepsin, as well as amylase type enzymes, lipase type enzymes, and mixtures thereof. Preferred enzymes include protease slurry, esperase slurry and amylase. A preferred enzyme is Esperse SL8 which is a protease. Anti-foam agents, e.g. silicon compounds, such as Silicane L 7604 can also be added in small effective amounts.

Bactericides, e.g. tetrachlorosalicylanilide and hexachlorophene, fungicides, dyes, pigments (water dispersible), preservatives, ultraviolet absorbers, anti-yellowing agents, such as sodium carboxymethyl cellulose, pH modifiers and pH buffers, colour safe bleaches, perfume, and dyes and bluing agents such as ultramarine blue can be used.

The composition may also contain an inorganic insoluble thickening agent or dispersant of very high surface area such as finely divided silica or extremely fine particle size (e.g. of 5-100 millimicrons diameters such as sold under the name Aerosil) or the other highly voluminous inorganic carrier materials disclosed in U.S.P. 3,630,929, in proportions of 0.1-10%, e.g. 1 to 5%. It is preferable, however, that compositions which form peroxyacids in the wash bath (e.g. compositions containing peroxygen compound and activator therefor) be substantially free of such compounds and of other silicates; it has been found, for instance, that silica and silicates promote the undesired decomposition of the peroxyacid.

In an embodiment of the invention the stability of the builder salts in the composition during storage and the dispersibility of the composition in water is improved by grinding and reducing the particle size of the solid builders to less than 100 microns, preferably less than 40 microns and more preferably to less than 10 microns. The solid builders, e.g. sodium tripolyphosphate (TPP), are generally supplied in particle sizes of about 100, 200 or 400 microns. The nonionic liquid surfactant phase can be mixed with the solid builders prior to or after carrying out the grinding operation.

In a preferred embodiment of the invention, the mixture of liquid nonionic surfactant and solid ingredients is subjected to an attrition type of mill in which the particle sizes of the solid ingredients are reduced to less than about 10 microns, e.g. to an average particle size of 2 to 10 microns or even lower (e.g. 1 micron). Preferably less than about 10%, especially less than about 5% of all the suspended particles have particle sizes greater than 10 microns. Compositions whose dispersed particles are of such small size have improved stability against separation or settling on storage. Addition of the acid terminated nonionic surfactant compound can decrease the yield stress of such dispersions and aid in the dispersibility of the dispersions without a corresponding decrease in the dispersion stability against settling.

In the grinding operation, it is preferred that the proportion of solid ingredients be high enough (e.g. at least about 40% such as about 50%) that the solid particles are in contact with each other and are not substantially shielded from one another by the nonionic surfactant liquid. After the grinding step any remaining liquid nonionic surfactant can be added to the ground formulation. Mills which employ grinding balls (ball mills) or similar mobile grinding elements have given very good results. Thus, one may use a laboratory batch attritor having 8 mm diameter steatite grinding balls. For larger scale work a continuously operating mill in which there are 1 mm or 1.5 mm diameter grinding balls working in a very small gap between a stator and a rotor operating at a relatively high speed (e.g. a CoBall mill) may be employed; when using such a

- mill, it is desirable to pass the blend of nonionic surfactant and solids first through a mill which does not effect such fine grinding (e.g. a colloid mill) to reduce the particle size to less than 100 microns (e.g. to about 40 microns) prior to the step of grinding to an average particle diameter below about 10 microns in the continuous ball mill.
- 5 In the preferred heavy duty liquid laundry detergent compositions of the invention, typical proportions (percent based on the total weight of composition, unless otherwise specified) of the ingredients are as follows:
- Liquid nonionic surfactant detergent in the range of about 10 to 60, such as 20 to 50 percent, e.g. about 30 to 40 percent;
- 10 Acid terminated nonionic surfactant may be omitted, it is preferred however that it be added to the composition in an amount in the range of about 2 to 20, such as 3 to 15 percent, e.g. about 4 to 10;
- Detergent builder, such as sodium tripolyphosphate (TPP), in the range of about 20 to 60, such as 25 to 45 percent, e.g. about 25 to 35;
- 15 Alkali metal silicate in the range of about 0 to 30, such as 5 to 25 percent, e.g. about 10 to 20;
- Copolymer of polyacrylate and polymaleic anhydride alkali metal salt anti-encrustation agent in the range of about 0 to 10, such as 2 to 8 percent, e.g. about 3 to 5;
- Alkylene glycol monoalkylether anti-gel agent may be omitted, it is preferred however that it be added to the composition in an amount in the range of about 5 to 20, such as 5 to 15 percent, e.g. about 8 to 12;
- 20 Higher alkyl sulphonate or higher alkyl poly lower alkoxy sulphate anti-settling agent in the range of 0.1 to 5, preferably 0.3 to 2.0 and more preferably about 0.5 to 1.5 percent; (It is an essential feature of the invention that at least one of the higher alkyl sulphonate or higher alkyl poly lower alkoxy sulphate anti-settling stabilizing agents be included in the composition);
- 25 Phosphoric acid alkanol ester stabilizing agent in the range of 0 to 2.0 or 0.1 to 2.0, such as 0.10 to 1.0 percent;
- Aluminium salt of fatty acid stabilizing agent in the range of about 0 to 5.0, such as 0.5 to 2.0 percent, e.g. about 0.1 to 1.0 percent;
- 30 Bleaching agent in the range of about 0 to 30, such as 2 to 20, e.g. about 5 to 15 percent; Bleach activator in the range of about 0 to 15, such as 1 to 10, e.g. about 2 to 6 percent; Sequestering agent for bleach in the range of about 0 to 3.0, preferably 0.5 to 2.0 e.g. about 0.75 to 1.25 percent;
- Anti-redeposition agent in the range of about 0 to 3.0, preferably 0.5 to 2.0 percent, e.g. 0.75 to 1.25 percent;
- 35 Optical brightener in the range of about 0 to 2.0, preferably 0.25 to 1.0 percent, e.g. 0.25 to 0.75 percent;
- Enzymes in the range of about 0 to 3.0, preferably 0.5 to 2.0 percent, e.g. 0.75 to 1.25 percent;
- 40 Perfume in the range of about 0. to 3.0, preferably 0.25 to 1.25 percent, e.g. 0.75 to 1.0 percent;
- Dye in the range of about 0 to 0.10, preferably 0.0025 to 0.050, e.g. 0.0025 to 0.0100 percent;
- Various of the previously mentioned additives can optionally be added to achieve the desired function of the added materials.
- 45 Mixtures of the acid terminated nonionic surfactant and the alkylene glycol alkyl ether anti-gel agents can be used and in some cases advantages can be obtained by the use of such mixtures alone, or with the addition to the mixture of a stabilizing and anti-settling agent.
- In the selection of the additives, they will be chosen to be compatible with the main constituents of the detergent composition. In this application, as mentioned above, all proportions and percentages are by weight of the entire formulation or composition unless otherwise indicated.
- 50 The concentrated nonaqueous nonionic liquid detergent composition of the present invention dispenses readily in the water in the washing machine. The presently used home washing machines normally use 250 gms of powder detergent to wash a full load of laundry. In accordance with the present invention only about 77 cc or about 100 gms of the concentrated liquid nonionic detergent composition is needed.
- 55 In a preferred embodiment of the invention the detergent composition of a typical formulation is formulated using the below named ingredients:

	Weight %	
Nonionic surfactant detergent.	30-40	
Acid terminated surfactant.	4-10	
5 Detergent builder salt.	25-35	5
Copolymer of polyacrylate and polymaleic anhydride alkali metal salt anti-encrustation agent (Sokalan CP-5).	3-5	
Alkylene glycol monoalkylether anti-gel agent.	8-12	10
10 Higher alkyl sulphonate sodium salt or higher alkyl polyethoxy sulphate sodium salt.	0.5-1.5	
Alkali metal perborate bleaching agent.	5-15	
15 Bleach activator (TAED).	2.0-6.0	15
Optical brightener (Stilbene Brightener N4).	0.25-0.75	
Enzymes (Protease-Esperease SL8).	0.75-1.25	
Perfume.	0.75-1.0	20
20		

The present invention is further illustrated by the following examples.

#### EXAMPLE 1

A concentrated nonaqueous liquid nonionic surfactant detergent composition is formulated from the following ingredients in the amounts specified.

	Weight %	
Plurafac RA 50 nonionic surfactant.	38.0	
Acid terminated Dobanol 91-5 reaction product with succinic anhydride.	6.0	30
Sodium tripolyphosphate.	34.0	
Diethylene glycol monobutyl ether anti-gel agent.	4.0	
C <sub>15</sub> Alkyl sulphonate sodium salt (Mersolat H98 C <sub>15</sub> ).	1.0	35
Sodium perborate monohydrate bleaching agent.	9.0	
Tetraacetylene diamine (TAED) bleach activator.	6.0	
Stilbene brightener.	0.8	
40 Protease (Esperease).	1.2	40

The addition of 1% of the alkyl sulphonate sodium salt is found to increase the yield stress of the formulation from about 2 to about 4 Pa. The viscosity of the formulation is found to remain about the same.

45 The formulation is ground for about 1 hour to reduce the particle size of the suspended builder salts to less than 40 microns. The formulated detergent composition is found to be stable and non-gelling in storage and to have a high detergent capacity.

#### EXAMPLE 2

50 A concentrated nonaqueous liquid nonionic surfactant detergent composition is formulated from the following ingredients in the amounts specified.

	Weight %	
	Plurafac RA 50 nonionic surfactant.	36.0
	Acid terminated Dobanol 91-5 reaction product	6.0
5	with succinic anhydride.	
	Sodium tri-polyphosphate (TPP).	32.0
	Diethylene glycol monobutylether anti-gel agent.	8.0
	Higher alkyl poly ethoxy ether sulphate sodium salt.	1.0
10		
	Sodium perborate monohydrate bleaching agent.	9.0
	Tetraacetylene diamine (TAED) bleaching agent.	5.0
	Stilbene brightener.	0.75
15	Protease (Esperase).	1.25
	Perfume.	1.0
		15

The formulation is ground for about one hour to reduce the particle size of the suspended builder salts to less than 40 microns. The formulated detergent composition is found to be stable and non-gelling in storage and to have a high detergent capacity.

The formulations of Examples 1 and 2 can be prepared without grinding the builder salts and suspended solid particles to a small particle size, but best results are obtained by grinding the formulation to reduce the particle size of the suspended solid particles.

The builder salts can be used as provided, or the builder salts and suspended solid particles can be ground or partially ground prior to mixing them with the nonionic surfactant. The grinding can be carried out in part prior to mixing and grinding completed after mixing or the entire grinding operation can be carried out after mixing with the liquid surfactant. The formulations containing suspended builder and solid particles less than 40 microns in size are preferred.

It is understood that the foregoing detailed description is given merely by way of illustration and that variations may be made therein without departing from the spirit of the invention.

#### CLAIMS

1. A fabric treating detergent composition which comprises a suspension of insoluble inorganic particles in a nonaqueous nonionic liquid surfactant detergent and an anionic higher alkyl sulphonate or higher alkyl polyether sulphate anti-settling agent to increase the stability of the suspension.

2. A composition as claimed in Claim 1 in which the higher alkyl sulphonate is a C<sub>8</sub> to C<sub>26</sub> primary or secondary alkyl sulphonate alkali metal salt.

3. A composition as claimed in Claim 1 or Claim 2 in which the higher alkyl sulphonate is a C<sub>14</sub> to C<sub>18</sub> primary alkyl sulphonate sodium salt.

4. A composition as claimed in Claim 1 in which the higher alkyl polyether sulphate is an alkali metal sulphate, the alkyl is C<sub>14</sub> to C<sub>20</sub> and the polyether contains 2 to 8 lower alkoxy groups.

5. A composition as claimed in Claim 1 in which the higher alkyl polyether sulphate is the sodium salt, the alkyl is C<sub>12</sub> to C<sub>15</sub> alkyl and the polyether contains two to four ethoxy groups.

6. A detergent composition as claimed in any one of Claims 1 to 5 in which the composition comprises at least one anti-gel agent selected from the group consisting of an acid terminated nonionic surfactant and an alkylene glycol monoalkyl ether.

7. A detergent composition as claimed in any one of Claims 1 to 6 comprising one or more detergent adjuvants selected from the group consisting of anti-incrustation agent, alkali metal silicate, bleaching agent, bleach activator, sequestering agent, anti-redeposition agent, optical brightener, enzymes, perfume and dye.

8. A detergent composition as claimed in any one of Claims 1 to 7 comprising 10 to 50 percent of an organic or inorganic builder salt.

9. A composition as claimed in Claim 8 in which the inorganic particles comprise an alkali metal polyphosphate detergent builder salt.

10. A composition as claimed in any one of Claims 1 to 9 in which the inorganic particles have a particle size distribution such that no more than about 10% by weight of the said particles have a particle size of more than about 10 microns.

11. A composition as claimed in any one of Claims 1 to 10 which contains from about 0.1 to about 5 percent by weight, based on the total composition, of said higher alkyl sulphonate or higher alkyl polyether sulphate.

12. A nonaqueous heavy duty, built laundry detergent composition which is pourable at high and low temperatures and does not gel when mixed with cold water, the said composition comprising

- at least one nonionic surfactant in an amount of from about 10 to about 60 percent by weight;
- at least one detergent builder suspended in the nonionic surfactant in an amount of from about 20 to about 60 per cent by weight;
- 5 an acid terminated nonionic surfactant as a gel inhibiting additive, in an amount up to about 2 to 20 percent by weight; 5
- a compound of the formula  $RO(CH_2CH_2O)_nH$
- where R is a  $C_2$  to  $C_8$  alkyl group and n is a number having an average value in the range of from about 1 to 6; as a gel inhibiting additive in an amount up to about 5 to 20 percent by weight; 10
- a higher alkyl sulphonate or a higher alkyl lower polyether sulphate anti-settling and stabilizing agent in an amount of about 0.3 to 2.0 percent by weight. 10
13. A detergent composition as claimed in Claim 12 which optionally contains, one or more detergent adjuvants selected from the group consisting of enzymes, corrosion inhibitors, anti-foam agents, suds suppressors, soil suspending or anti-redeposition agents, anti-yellowing agents, colourants, perfumes, optical brighteners, bluing agents, pH modifiers, pH buffers, bleaching agents, bleach stabilizers, bleach activators, enzyme inhibitors and sequestering agents. 15
14. A nonaqueous liquid heavy duty laundry detergent composition as claimed in Claim 12 or Claim 13 which comprises 20
- |  |            |    |
|--|------------|----|
|  | Weight %   |    |
| Nonionic surfactant in an amount of about  | 30-40      |    |
| Acid Terminated surfactant in an amount of about                                     | 4-10       |    |
| 25 Sodium tripolyphosphate (TPP) in an amount of about                               | 25-35      | 25 |
| Copolymer of polyacrylate and polymaleic anhydride sodium salt in an amount of about | 3-5        |    |
| 30 Diethylene glycol monobutylether in an amount of about                            | 8-12       | 30 |
| $C_{14}$ to $C_{16}$ alkyl sulphonate sodium salt in an amount of about              | 0.3 to 2.0 |    |
| Sodium perborate monohydrate bleaching agent in an amount of about                   | 8-12       | 35 |
| Tetraacetylene diamine (TAED) bleach activator in an amount of about                 | 3.5-5.5    |    |
15. A nonaqueous liquid heavy duty laundry detergent composition as claimed in Claims 12, 13 or 14 which comprises 40
- |  |            |    |
|--|------------|----|
|  | Weight %   |    |
| Nonionic surfactant in an amount of about  | 30-40      |    |
| 45 Acid Terminated surfactant in an amount of about                                  | 4-10       | 45 |
| Sodium tripolyphosphate in an amount of about  | 25-35      |    |
| Copolymer of polyacrylate and polymaleic anhydride sodium salt in an amount of about | 3-5        | 50 |
| 50 Diethylene glycol monobutylether in an amount of about                            | 8-12       |    |
| $C_{12}$ to $C_{14}$ alkyl poly ethoxy sulphate sodium salt in an amount of about    | 0.3 to 2.0 |    |
| 55 Sodium perborate monohydrate bleaching agent in an amount of about                | 8-12       | 55 |
| Tetraacetylene diamine (TAED) bleach activator in an amount of about                 | 3.5-5.5    |    |
- 60 16. A composition as claimed in Claim 1, substantially as specifically described herein with reference to Example 1 or Example 2. 60
17. A method for cleaning soiled fabrics which comprises contacting the soiled fabrics with the laundry detergent composition as claimed in any one of Claims 1 to 16.



---

Published 1988 at The Patent Office, State House, 66/71 High Holborn, London WC1R 4TP. Further copies may be obtained from The Patent Office, Sales Branch, St Mary Cray, Orpington, Kent BR5 3RD. Printed by Burgess & Son (Abingdon) Ltd. Con. 1/87.